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## INTERNATIONAL APPLICATION PUBLISHED UNDER THE PATENT COOPERATION TREATY (PCT)

(51) International Patent Classification 6:

(11) International Publication Number:

WO 99/06611

C23C 16/44

A1

(43) International Publication Date:

11 February 1999 (11.02.99)

(21) International Application Number:

PCT/US98/15706

(22) International Filing Date:

29 July 1998 (29.07.98)

(81) Designated States: JP, KR, European patent (AT, BE, CH, CY, DE, DK, ES, FI, FR, GB, GR, IE, IT, LU, MC, NL, PT,

SE)

**Published** 

(30) Priority Data:

08/904,656

1 August 1997 (01.08.97)

us |

With international search report.

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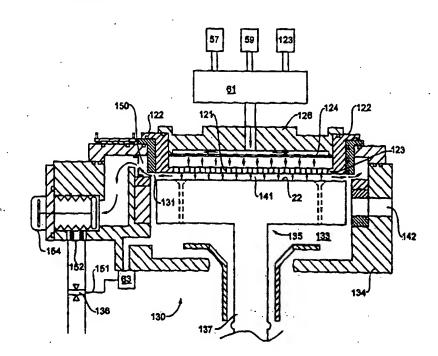
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(54) Title: METHOD AND APPARATUS FOR CHAMBER CLEANING

#### (57) Abstract

method apparatus for cleaning a processing chamber. Steps of the method include flowing a cleaning gas into the chamber, and flowing an inert gas into the chamber during at least a portion of the same time as the cleaning gas, such that the ratio of inert gas to cleaning gas is in a range of about 1:1 to about 1:4 by volume. The apparatus (130)includes a sensor (63) for measuring total pressure in the interior of the chamber (133). Two cleaning gas supplies (59) may be used: a cleaning gas supply (59) with a first valved inlet providing an entrance to the interior of the chamber for passing cleaning gas to the interior of the chamber, and an inert gas supply (57) with a second valved inlet providing an entrance to the



interior of the chamber for passing an inert gas to the interior of the chamber. A governor (136) with an input coupled to the sensor maintains the total pressure within the chamber at a prespecified value. First and second mass flow controllers are coupled to the first and second valved inlets, such that the first and second mass flow controllers are set to control the ratio of the amount of cleaning gas entering the chamber to the amount of inert gas entering the chamber so that this ratio is maintained in a range of about 1:1 to about 1:4.

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#### METHOD AND APPARATUS FOR CHAMBER CLEANING

#### **BACKGROUND OF THE INVENTION**

The present invention relates generally to chemical vapor deposition (CVD) processing, and more particularly to a method and apparatus for CVD chamber cleaning.

CVD is widely used in the semiconductor industry to deposit films of various kinds, such as intrinsic and doped amorphous silicon (a-Si), silicon oxide (Si<sub>x</sub>O<sub>y</sub>), silicon nitride (Si<sub>r</sub>N<sub>s</sub>), silicon oxynitride, and the like on a substrate. Modern semiconductor CVD processing is generally done in a vacuum chamber by using precursor gases which dissociate and react to form the desired film. In order to deposit films at low temperatures and relatively high deposition rates, a plasma can be formed from the precursor gases in the chamber during the deposition. Such processes are known as plasma-enhanced CVD processes (PECVD).

State of the art CVD semiconductor processing chambers are made of aluminum and include a support for the substrate and a port for entry of the required precursor gases. When a plasma is used, the gas inlet and/or the substrate support is connected to a source of power, such as a radio frequency (RF) power source. A vacuum pump is also connected to the chamber to control the pressure in the chamber and to remove the various gases and particulates generated during the deposition.

In all semiconductor processing, particulates in the chamber must be kept to a minimum. Particulates are formed because, during the deposition process, the film is deposited not only on the substrate, but also on walls and various fixtures, e.g., shields, the substrate support and the like, in the chamber. During subsequent depositions, the film on the walls, etc., can crack or peel, causing contaminant particles to fall on the substrate. This causes problems and damage to particular devices on the substrate. Damaged devices have to be discarded.

When large glass substrates, e.g., of sizes up to 360 mm x 450 mm or even larger, are processed to form thin film transistors for use as computer screens and the like, more than a million transistors may be formed on a single substrate. The presence of contaminates in the processing chamber is even more serious in this case, since the computer screen and the like will be inoperative if damaged by particulates. In this case, an entire large glass substrate must be discarded.

Thus, the CVD chamber must be periodically cleaned to remove accumulated films from prior depositions. Cleaning is generally done by introducing an etch gas, such as a fluorine-containing gas, e.g., nitrogen trifluoride (NF<sub>3</sub>), into the chamber. A plasma is initiated from the fluorine-containing gas which reacts with coatings from prior depositions on the chamber walls and fixtures, e.g., coatings of a-Si, Si<sub>x</sub>O<sub>y</sub>, Si<sub>r</sub>N<sub>s</sub>, SiON and the like, as well as any other materials in the chamber. In particular, the NF<sub>3</sub> creates free fluorine radicals "F\*" which react with Si-containing residues.

The reaction forms gaseous fluorine-containing volatile products that can be pumped away through the chamber exhaust system. This procedure is generally followed by a nitrogen purge.

As the volatiles are pumped away, their contribution to the overall pressure stays low until there are no residues left with which the F\* can react. Then their contribution to the overall pressure, and thus the overall pressure itself, rises. One can use this rise to detect the endpoint of the cleaning procedure by monitoring the overall pressure with a manometer and waiting for a user-defined endpoint pressure to be reached.

This technique is vulnerable to, e.g., variations in the manometer pressure reading over time, i.e., "manometer pressure drift". Such variations are inherent in any such instrument and may be typically provided for with appropriate calibration techniques. If the variation is such that the manometer drifts low, the process may not seem to attain the endpoint. In this situation, the cleaning recipe is carried on indefinitely, or until a preset time is reached, termed a "recipe timeout". Even though the chamber is cleaned,

throughput is reduced: Furthermore, with NF<sub>3</sub>, cost is increased because NF<sub>3</sub> is significantly more expensive than other process gases, and flowing a large and constant amount of NF<sub>3</sub> is costly and inefficient.

In the opposite case, if the manometer drifts high, the preset pressure may be attained prior to endpoint, resulting in an incomplete clean. Here, films may be left in the chamber that are a potential source of contaminant particles.

#### SUMMARY OF THE INVENTION

In one aspect, the invention is directed to a method for cleaning a processing chamber. Steps of the method include flowing a cleaning gas into the chamber, and flowing an inert gas into the chamber during at least a portion of the same time as the cleaning gas, such that the ratio of inert gas to cleaning gas is in a range of about 1:1 to about 1:4 by volume.

Implementations of the invention may include one or more of the following. The cleaning gas may be NF<sub>3</sub>, and the inert gas may be Ar. The NF<sub>3</sub> may be flowed at a rate between about 6.7 and 13.4 standard cubic centimeters per second per liter of chamber volume, such as about 12.2 standard cubic centimeters per second per liter of chamber volume. The Ar may be flowed at a rate of between about 11.1 and 44.4 standard cubic centimeters per second per liter of chamber volume.

In another aspect, the invention is directed to a cleaning system for a processing chamber. The system includes a sensor for measuring total pressure in the interior of the chamber. Two cleaning gas supplies may be used: a cleaning gas supply with a first valved inlet providing an entrance to the interior of the chamber for passing cleaning gas to the interior of the chamber, and an inert gas supply with a second valved inlet providing an entrance to the interior of the chamber for passing an inert gas to the interior of the chamber. A governor with an input coupled to the sensor maintains the total pressure within the chamber at a prespecified value. First and second mass flow controllers are coupled to the first and second valved inlets, such that

the first and second mass flow controllers are set to control the ratio of the amount of cleaning gas entering the chamber to the amount of inert gas entering the chamber so that this ratio is maintained in a range of about 1:1 to 1:4. The pressure sensor may be a manometer or an ion gauge.

An advantage of the invention is that a significantly reduced amount of cleaning gas is necessary to clean a chamber. Less waste of costly NF<sub>3</sub> occurs. Thus, the cleaning process is less expensive. A further advantage is that deposited materials may be removed from walls of a reaction chamber at rates comparable to those using other processes, thus throughput is not sacrificed.

## BRIEF DESCRIPTION OF THE DRAWING

Figure 1 is a cross-sectional plan view of a PECVD chamber useful for depositing thin films on a substrate which may be used in accordance with an embodiment of the present invention.

#### **DETAILED DESCRIPTION**

US Patent 5,366,585, issued November 22, 1994 to Robertson *et al.*, entitled "Rapid Thermal Heating Apparatus and Method", assigned to the assignee of the present invention, and incorporated herein by reference, discloses a PECVD chamber which may be cleaned using an embodiment of the present invention. Of course, the invention is applicable to numerous other types of CVD devices as well as devices for other deposition methods. For example, the invention may be used in chambers which carry out the following fabrication processes: CVD, PECVD, thermally enhanced CVD (TECVD), etc. Certain of the details described are specific to this implementation and may be changed as required by the processing conditions and parameters.

The present invention may be used in AKT PECVD systems manufactured by Applied Komatsu Technology of Santa Clara, California. For example, the AKT-3500 PECVD

is designed for use in the production of substrates for large liquid crystal flat panel displays. It is a modular system with multiple process chambers which can be used for depositing a-Si, Si<sub>r</sub>N<sub>s</sub>, Si<sub>x</sub>O<sub>y</sub>, oxynitride films, and other similar films. The present invention, however, may be used with any commercially-available deposition system.

PECVD or CVD processes used to deposit a thin film layer onto a substrate. We intend the term "substrate" to broadly cover any object that is being processed in a process chamber. The term "substrate" includes, for example, semiconductor wafers, flat panel displays, and glass plates or disks. The present invention is particularly applicable to large substrates such as glass plates having areas of 360 x 450 mm, 550 x 650 mm, and larger. The remainder of this detailed description describes an embodiment in which a glass substrate is used. However, as noted above, other substrates may also be used.

In general, the substrate is supported in a vacuum deposition process chamber, and the substrate is heated to several hundred degrees Celsius (°C). Deposition gases are injected into the chamber, and a plasma-enhanced chemical reaction occurs to deposit a thin film layer onto the substrate. The thin film deposited layer may be a dielectric layer (such as SiN or SiO), a semiconductor layer (such as a-Si) or a metal layer (such as tungsten(W)).

In the chamber shown in Figure 1, a plasma is employed to enhance deposition. Accordingly, appropriate plasma ignition means, such as the RF voltage described below, are generally required. Alternatively, a remote plasma source (not shown) may be used to provide the plasma. More details of a remote plasma chamber may be found in U.S. Patent Application Serial No. 08/707,491, entitled "A Deposition Chamber Cleaning Technique Using a High Power Remote Excitation Source", filed January 14, 1997, assigned to the assignee of the present invention and incorporated herein by reference.

As shown in Figure 1, a CVD apparatus 130 includes a susceptor 135 having a stem 137. Susceptor 135 is centered within a vacuum deposition process chamber 133. Susceptor 135 holds a substrate such as a glass panel (not shown) in a substrate

processing or reaction region 141. A lift mechanism (not shown) is provided to raise and lower the susceptor 135. Commands to the lift mechanism are provided by a controller. Substrates are transferred into and out of chamber 133 through an opening 142 in a sidewall 134 of the chamber 133 by a robot blade (not shown).

The deposition process gases flow into chamber 133 through a manifold 61 and an inlet 126. The gases then flow through a perforated blocker plate 124 and a number of holes 121 in a process gas distribution faceplate 122 (indicated by small arrows in the substrate processing region 141 of Figure 1). An RF power supply (not shown) may be used to apply electrical power between gas distribution faceplate 122 and susceptor 135 so as to excite the process gas mixture to form a plasma. The constituents of the plasma react to deposit a desired film on the surface of the substrate on susceptor 135.

The deposition process gases may be exhausted from the chamber through a slot-shaped orifice 131 surrounding substrate processing region 141 into an exhaust plenum 150. From exhaust plenum 150, the gases flow by a vacuum shut-off valve 154 and into an exhaust outlet 152 which connects to an external vacuum pump (not shown).

Chamber 133 has at least two other gas supplies: an argon (Ar) supply 57 and an NF<sub>3</sub> supply 59. These gases are used for the cleaning procedure.

A manometer 63 measures the total pressure of gases in chamber 133. Of course, manometer 63 could be replaced by numerous other types of sensors for total pressure with equally good results. As an example, an ion gauge could be used.

A governor 136 is disposed in the exhaust stream to regulate the overall pressure as measured by manometer 63. In particular, a signal 151 from manometer 63 may be used as an input to an electrical controller of governor 136 so as to keep the total pressure constant.

Chamber 133 may be cleaned by establishing a flow of gas into chamber 133 and creating a plasma therefrom. In particular, in this embodiment, a flow of both NF<sub>3</sub> and

Ar is established and the system is brought to a temperature such as that used for deposition which may be about 350 degrees C to 400 degrees C. The gas flow may produce a range of total pressures in the chamber of about 0.1 Torr to about 2.0 Torr, and more particularly about 1 Torr  $\pm 1/2$  Torr with a maximum pressure of about 10 Torr. Generally, if too high a pressure is used, the resulting plasma will be unstable. If too low a pressure is used, the cleaning rate will be too low. A susceptor-gas manifold spacing of about 500 mil to 2500 mil may be employed.

The plasma of gases may be formed either in-situ in the chamber or ex-situ in a remote plasma chamber. In the latter embodiment, gases from the remote plasma chamber enter chamber 133 from a separate valved inlet as described in the patent application incorporated by reference above.

The present invention conserves NF<sub>3</sub> by the use of a governor in the chamber exhaust port. The governor varies exhaust conductance in such a manner as to keep the pressure of NF<sub>3</sub> constant within the chamber. In this way, the NF<sub>3</sub> flow rate can be greatly reduced for the same etching rate, and the NF<sub>3</sub> gas is fully utilized in the chamber, eliminating wasted NF<sub>3</sub>. In such a system, the endpoint of the cleaning procedure is not generally detected using the rise of the chamber total pressure, since the total pressure is by design constant.

The present invention can use a low flow rate of NF<sub>3</sub>, such as from about 300 to 800 sccm, e.g., about 550 sccm. The other gas, which may be, like Ar, an inert gas, is also flowed into the chamber simultaneously at a flow rate of from about 500 to 2000 sccm, for example 1100 sccm. Of course, such rates depend on the size of the chamber to be cleaned. Generally, NF<sub>3</sub> rates such as about 6.7 sccm to 13.4 sccm per liter of chamber volume may be appropriate (such as about 12.2 sccm per liter), as may Ar rates of about 11.1 sccm to 44.4 sccm per liter of chamber volume. Similarly, NF<sub>3</sub> rates such as 0.15 sccm to 0.4 sccm per square centimeter of substrate area would be appropriate, as would Ar rates of 0.3 sccm to 1.2 sccm per square centimeter of substrate area. Generally, the rates used should result in a flow rate ratio of NF<sub>3</sub>:Ar in a range of between about 1:1 to 1:4.

Alternatively, and for a typical value of conductance of the governor, the flow rates may result in predetermined partial pressures for the cleaning gas and for the inert gas, e.g., for NF<sub>3</sub> and for Ar. These partial pressures may be estimated to first order, in known manner from variables including the flow rates and the overall pressure.

An RF power of about 1000 watts to about 4000 watts is applied to the gas manifold to produce a plasma, corresponding to power densities per substrate processing area of about 0.5 watts/cm<sup>2</sup> to about 3 watts/cm<sup>2</sup>. Different deposited materials vary in the rates in which they may be cleaned or removed. For example, the plasma cleans at a rate of about 3000 angstroms per minute to about 4000 angstroms per minute for a-Si, and at a rate of about 6000 angstroms per minute to about 7500 angstroms per minute for SiN.

The plasma of NF<sub>3</sub> and Ar produces a desirable cleaning effect. The chemical mechanisms responsible for the cleaning effect are not well understood. However, NF<sub>3</sub> diluted with Ar generally dissociates more completely than undiluted NF<sub>3</sub>.

Adding an inert, noble or molecular gas to an NF<sub>3</sub> plasma has additional advantages. For example, the discharge is more stable. One reason for this may be that excessive fluorine ion concentrations present in undiluted NF<sub>3</sub> reduce the free electron concentration. A reduction in the free electron concentration is associated with unstable plasmas. Plasma stability is generally desirable and often requires balanced process parameters. These process parameters include gas flow rates, power, pressure, and spacing. Direct relationships exist between the pressure, the free electron temperature, the RF power dissipation, and the free electron concentration. Such relationships may be found in, e.g., <u>Plasma Processing in Semiconductor Electronics</u>, CRC Press, incorporated by reference herein.

At least two techniques may be used to determine when the above method has finished cleaning the chamber. First, an optoelectronic endpoint detector may be used, as described in U.S. Patent Application entitled "Method and Apparatus for Detecting the

Endpoint of a Chamber Cleaning", to W. Blonigan and J.T. Gardner, filed on even date herewith, assigned to the assignee of the present invention, and incorporated herein by reference. Second, if the types and amounts of deposited materials are known, as well as the corresponding etching rates, the overall time of cleaning may be monitored. In this technique, once an adequate period of time has passed for all the deposited materials to be etched, the cleaning is terminated.

The above embodiment has been described with respect to certain gases. In particular, Ar has been used as the inert gas and NF<sub>3</sub> the cleaning gas. Other inert, noble and molecular gases may also be used, such as helium, neon, krypton, xenon, and molecular nitrogen, as well as other gases with similar properties. Moreover, other cleaning gases may also be used, such as CF<sub>4</sub>, C<sub>2</sub>F<sub>6</sub>, and SF<sub>6</sub>, as well as other gases with similar cleaning properties. Often these gases will contain fluorine.

The cleaning method may be used to clean a number of deposited films. For example, films of Si<sub>x</sub>O<sub>y</sub>, Si<sub>r</sub>N<sub>s</sub>, a-Si, polysilicon and SiON may all be removed by the invention.

The present invention has been described in terms of a preferred embodiment. The invention, however, is not limited to the embodiment depicted and described. Rather, the scope of the invention is defined by the appended claims.

What is claimed is:

#### **CLAIMS**

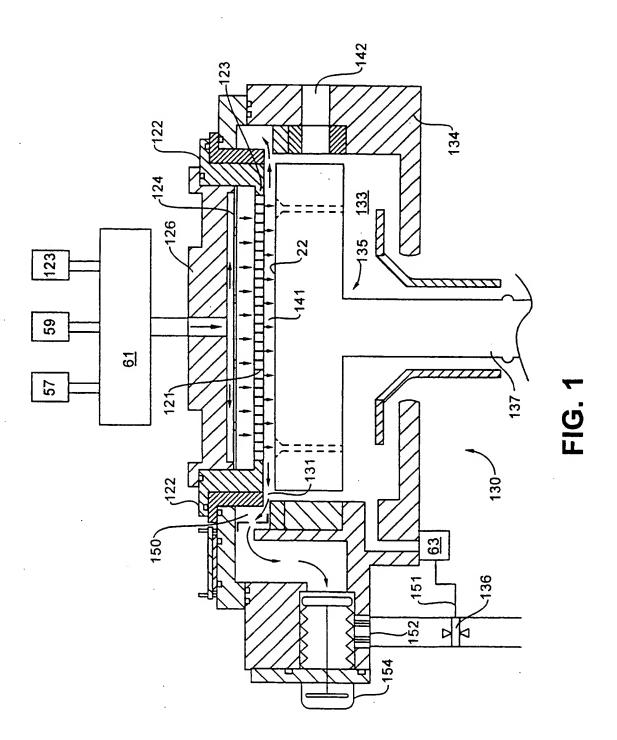
1. A method for cleaning a processing chamber, comprising the steps of:

- (a) flowing a cleaning gas into the chamber:
- (b) flowing an inert gas into the chamber during at least a portion of the same time as the cleaning gas, such that the ratio of inert gas to cleaning gas is in a range of about 1:1 to about 1:4 by volume.
- 2. The method of claim 1, wherein the cleaning gas is NF<sub>3</sub>.
- 3. The method of claim 2, wherein the inert gas is Ar.
- 4. The method of claim 3, wherein the NF<sub>3</sub> is flowed at a rate between about 6.7 and 13.4 standard cubic centimeters per second per liter of chamber volume.
- 5. The method of claim 4, wherein the Ar is flowed at a rate of between about 11.1 and 44.4 standard cubic centimeters per second per liter of chamber volume.
- 6. The method of claim 5, wherein the NF<sub>3</sub> is flowed at a rate of about 12.2 standard cubic centimeters per second per liter of chamber volume.
- 7. A cleaning system for a processing chamber, comprising:
  - a sensor for measuring total pressure in the interior of the chamber;
- a cleaning gas supply with a first valved inlet providing an entrance to the interior of the chamber for passing cleaning gas to the interior of the chamber;
- an inert gas supply with a second valved inlet providing an entrance to the interior of the chamber for passing an inert gas to the interior of the chamber;
- a governor with an input coupled to said sensor for maintaining the total pressure within the chamber at a prespecified value; and

first and second mass flow controllers coupled to said first and second valved inlets,

such that the first and second mass flow controllers are set to control the ratio of the amount of cleaning gas entering the chamber to the amount of inert gas entering the chamber so that this ratio is maintained in a range of about 1:1 to 1:4.

- 8. The system of claim 7, wherein the cleaning gas is NF<sub>3</sub>.
- 9. The system of claim 8, wherein the inert gas is Ar.
- 10. The system of claim 7, wherein said pressure sensor is a manometer.
- 11. The system of claim 7, wherein said pressure sensor is an ion gauge.



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# INTERNATIONAL SEARCH REPORT

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	ENTS CONSIDERED TO BE RELEVANT				
Category *	Citation of document, with indication, where appropriate, of the rel	evant passages	Relevant to claim No.		
X	US 5 413 670 A (LANGAN JOHN G E 9 May 1995 see abstract; claims 1,7,8; figur		1-11		
X	EP 0 731 497 A (AIR PROD & CHEM SYSTEMS INC (US); GEC INC (US)) 11 September 1996 see claim 1		1-6		
<b>X</b>	EP 0 638 923 A (APPLIED MATERIALS 15 February 1995 see page 4, line 44 - line 48; c' tables I,II	•	1-6		
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. national Application No PCT/US 98/15706

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	tion) DOCUMENTS CONSIDERED TO BE RELEVANT	•
Category *	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
Х	US 5 637 153 A (NIINO REIJI ET AL) 10 June 1997 see column 8, line 21 - column 9, line 18;	1
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## INTERNATIONAL SEARCH REPORT

Information on patent family members

PCT/US 98/15706

Patent document cited in search report	Publication date	Patent family member(s)	Publication date
US 5413670	A 09-05-1995	JP 2690460 B JP 7153739 A	10-12-1997 16-06-1995
EP 0731497	A 11-09-1996	US 5714011 A JP 8255778 A SG 33639 A	03-02-1998 01-10-1996 18-10-1996
EP 0638923	A 15-02-1995	US 5421957 A EP 0857796 A JP 7078808 A	06-06-1995 12-08-1998 20-03-1995
US 5637153	A 10-06-1997	JP 7297127 A US 5380370 A	10-11-1995 10-01-1995